K 96/116 M 96/117

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	5 Nov. '96	Final Tech 15 May '93 to 14 May '96

4. TITLE AND SUBTITLE
Atomic Mechanisms of Flow and Fracture at Metal-Ceramic Interfaces and Their Role in the Design of Metal Matrix Composites

5. FUNDING NUMBERS
F49620-93-1-0228

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8. PERFORMING ORGANIZATION REPORT NUMBER
AFOSR-TR-97
0044

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10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

DISTRIBUTION STATEMENT A
Approved for public release
Distribution Unlimited

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

This report describes new and significant results that can be applied in the microstructure design for optimum mechanical performance of metal-ceramic composites and laminates. There are three elements to these recommendations: (i) the design of the atomic structure of metal-ceramic interfaces, (ii) identification of the critical length scale in the two phase microstructure, and (iii) prediction of the microstructural conditions under which the thermal conductivity of the composite becomes significantly influenced by the thermal boundary resistance of interfaces. In the first topic we show that the beneficial effect of titanium interlayers at a copper/alumina interface is accomplished with only about one monolayer; with further increase in the titanium interlayer thickness having an insignificant effect on the interfacial strength. In the second topic we show that the metal ligament size is the key microstructural parameter in controlling the flow stress, the fracture stress and the fracture toughness of metal-ceramic composites. The metal ligament size is important because dislocation activity in the metal, which produces pile ups against the interface, is the critical event in flow and fracture of composites. In the third area we show that the interfacial thermal boundary resistance plays a dominant role in the overall thermal conductivity of the composite when the microstructural scale becomes smaller than about 1um.

14. SUBJECT TERMS
Interfaces, MMC, Intermetallics, Oxidation, Grain Boundary Sliding, Composites, Fracture

15. NUMBER OF PAGES
24

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
U	U	U	U

NSN 7540-01-280-5500

Standard Form 298 (Rev 2-89)
Prescribed by ANSI Std 239-18
298-102

DTIC QUALITY INSPECTED 1

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Abstract

This report describes new and significant results that can be applied in the microstructure design for optimum mechanical performance of metal-ceramic composites and laminates. There are three elements to these recommendations: (i) the design of the atomic structure of metal-ceramic interfaces, (ii) identification of the critical length scale in the two phase microstructure, and (iii) prediction of the microstructural conditions under which the thermal conductivity of the composite becomes significantly influenced by the thermal boundary resistance of interfaces. In the first topic we show that the beneficial effect of titanium interlayers at a copper/alumina interface is accomplished with only about one monolayer; with further increase in the titanium interlayer thickness having an insignificant effect on the interfacial strength. In the second topic we show that the metal ligament size is the key microstructural parameter in controlling the flow stress, the fracture stress and the fracture toughness of metal-ceramic composites. The metal ligament size is important because dislocation activity in the metal, which produces pile ups against the interface, is the critical event in flow and fracture of composites. In the third area we show that the interfacial thermal boundary resistance plays a dominant role in the overall thermal conductivity of the composite when the microstructural scale becomes smaller than about 1μm.

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Nov/5/96

Dr. C. H. Ward
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Final Technical Report: FQ8671-9300912

Dear Dr. Ward:

I am enclosing six copies of the final technical report for the above AFOSR grant on Metal-Ceramic Interfaces.

There were three notable achievements in the project:

(a) We identified the metal ligament size as the critical length scale in the influence of microstructure on the flow, fracture and creep properties of two phase architectures constructed from metals and ceramics, including composites and laminates.

(b) We demonstrated a new nanoengineering concept for enhancing interfacial bonding at metal-ceramic interfaces. Through high controlled experiments we showed that just a few monolayers of titanium is enough to significantly increase bonding at copper-sapphire interface. Thicker layers of titanium did not produce a further increase in interfacial bonding.

(c) From controlled experiments and theory we have identified a length parameter which describes the influence of interfaces on the thermal conductivity of metal ceramic composites and laminates. This parameter which we call the Kapitza Radius, has been found to have a relatively constant number of about 1μm in three different bimaterials. The theoretical back up shows that interfaces become a significant factor in the overall thermal conductivity of composites when the length scale of the microstructure becomes smaller than the Kapitza Radius. For example, in particulate composites, the thermal conductivity will be particle size dependent when the particle size becomes *smaller* than 1μm.

Please do not hesitate to call if you need copies of reprints or preprints which are described in the report. We appreciate the support from AFOSR in the above research.

Yours sincerely,

Rishi Raj

Rishi Raj

Refereed Publications

In this final report we give a perspective of the most significant accomplishments from the work supported by the AFOSR grant. The details of the results are given in the following papers which are either already in print or have been accepted for publication:

P1 R. Raj, "Single Crystal Based Microstructure Design of Metal Matrix Composites for High Temperature Applications", Critical Issues in the Development of High Temperature Structural Materials, eds. N. S. Stoloff, D. J. Duquette and A. F. Giamei, TMS, Warrendale PA, 1993, pp 71-86.

P2 "*In-situ* Stress-Strain Response of Small Metal Particles Embedded in a Ceramic Matrix", L. R. Thompson and R. Raj, Acta Metallurgica et Materialia, 42[7], 2477-2485 (1994).

P3 "Design of the Microstructural Scale for Optimum Toughening in Metallic Composites", R. Raj and L. R. Thompson, Acta Metallurgica et Materialia, 42[12], 4135-4142 (1994).

P4 "The Influence of Microstructural Scale on the Creep Resistance of High Volume Fraction Ceramic-Metal Composites made from Aluminum Oxide and Niobium", Y. Wang and R. Raj, Mater. Sci. and Eng., A206 128-137 (1996).

P5 "A Microindentation Method for Estimating Interfacial Shear Strength and Its Use in Studying the Influence of Titanium Transition Layers on Interface Strength of Epitaxial Copper Films on Sapphire", G. Dehm, M. Rühle, H.D. Conway and R. Raj, Acta Materialia, in press (1996-97).

P6 "The Influence of the Microstructural Scale on the Plastic Flow Behavior of Metal Matrix Composites", T. W. Gustafson, P.C. Panda, G. Song and R. Raj, Acta Materialia, in press (1996-97).

P7 "Growth and Structure of Internal Cu/Al_2O_3 and $Cu/Ti/Al_2O_3$ Interfaces", G. Dehm, C. Scheu, M. Rühle, and R. Raj, Acta Materialia, in press (1996-97).

P8 "Thermal Diffusivity of Particulate Composites made from Aluminum Oxide and Nickel Aluminide by a Photothermal Deflection Technique", Y. D. Chung, A. P. Chojnacka, C. T. Avedisian and R. Raj, Acta Materialia, in press (1996-97).

Personnel: Among the coauthors of the above papers, the graduate students, who have completed the Ph.D. or the M.S. degrees at Cornell University are:

L. R. Thompson (Ph.D.)
Y. Wang (M.S.)
T. W. Gustafson (Ph.D.)
A. P. Chojnacka (current Ph.D. student)

G. Dehm completed his Ph.D. at the Max Planck Institute for Metallurgy in Stuttgart, Germany.

Summary of Accomplishments

The accomplishments in the project may be divided into three sections. These three areas and the theme of the research in each of them is given below. Following that, the highlights in each area are discussed in greater detail.

(a) The first area is concerned with dislocation mechanisms for explaining *interface controlled* deformation and fracture of high volume metal-matrix composites, at ambient and at high temperature. The objective of these studies was to identify the principal microstructural scale that is the dominant factor in the mechanical performance of the composite. Detailed experimental and modelling work has identified the size of the metal ligament that lies between adjacent metal/ceramic interfaces, as the critical length scale in the microstructure. *These results are described in publications P2, P3, P4 and P6 in the list given above.*

(b) The second area is concerned with the use of nanoengineering of metal-ceramic interfaces in order to improve the mechanical properties of the interface. We have shown that just one monolayer of titanium can increase the interfacial strength of a copper-alumina interface by a factor of 1.5-2.0. Thicker interlayers of titanium did not produce a further meaningful increase in the interfacial strength.

These results are discussed in papers P5 and P7.

(c) The third area is concerned with the role of interfaces in affecting the thermal conductivity of metal-ceramic composite. Modelling and experiments have led to the definition of a new parameter, which we call the Kapitza Radius, r_k. This parameter is related to the *thermal boundary resistance* of the interface, but is normalized in such a way that it expresses the thermal resistance as a length scale. The important result of the work is that the interfacial resistance becomes important when the microstructural scale of the dispersed phase is smaller than r, thus leading to a simple way of predicting when thermal conductivity of composites and laminates will be dominated by interfaces. *These results are given in publication P8.*

In the following sub-sections these three areas are discussed in greater detail by giving examples of experimental and theoretical results.

(a) Interface Controlled Dislocation Mechanisms for Creep, Deformation and Fracture in Metal Matrix Composites.

(a.1) DEFORMATION AND FRACTURE OF METAL MATRIX COMPOSITES

The work we have performed in this project investigates how the microstructural scale can control high temperature creep, yield strength and the fracture toughness of metal matrix composites. Our objective was the search for a critical length scale in the microstructure, in the same vein as the Hall-Petch

equation highlights the importance of the grain size in yield behavior of single phase polycrystalline materials.

In metal matrix composites, the microstructure is ostensibly more complex. The variables that define the geometrical properties of the microstructure include: the particle size of the dispersed (ceramic) phase, the volume fraction of the ceramic phase, and the length scale of the continuous phase (usually a metal). The last parameter is equal to the width of the metal phase confined between two adjacent metal-ceramic interfaces: it may be described as the metal ligament size (see P6). In practice only two of these three variables are independent. For example, for the fixed volume fraction, a change in the particle size causes a corresponding change in the metal ligament size. With the assumption that the particles have the shape of cubes, we can show that[1]:

$$\lambda_m = a\left(\frac{1}{f_v^{1/3}} - 1\right) \qquad \text{(P6-1)}$$

where λ_m is the metal ligament size, f_v is the volume fraction of the dispersed phase, and a is the particle size of the dispersed phase.

The importance of length scales in mechanical behavior is revealed only by considering the defect mechanisms that control deformation and fracture. Continuum models are sensitive only to the volume fraction of the second phase. For example the inverse

[1]The first two symbols in the equation number refer to the publication number, P1, P2, etc.

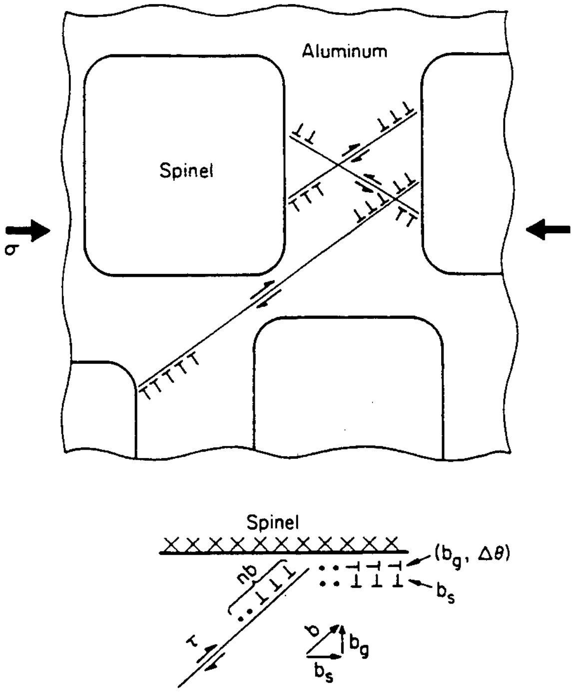
square root dependence of the yield strength on the grain size in the Hall-Petch equation is explained by considering the yield behavior to be controlled by discreet events such as the pile of dislocations at grain boundaries.

In the present work we have developed a a new concept for the movement of dislocations in a Metal Matrix Composite. We invoke slip compatibility at the metal-ceramic interface as the key step in the deformation and fracture of the composite. The concept is illustrated in Figs. 1(a) and 1(b). Case (a) refers to uniaxial compression and case (b) to uniaxial tension. The change in the stress state increases the likelihood of plastic flow in compression and fracture in tension. In plastic flow, case (a), sliding at the metal-ceramic interfaces is accommodated by dislocations gliding parallel to the interface, shown by the Burgers vector b_s; since the slip vector is at an angle the Burgers vector of the incident dislocation, b, decomposes into b_s and b_g, the first parallel and the second normal to the interface. The residual dislocations with Burgers vector b_g create a low angle boundary. *The creation of these low angle boundaries was confirmed by TEM studies of deformed specimens (see Ref. P6)*. The low angle boundaries create a back stress which then controls further deformation. The <u>lower bound</u> on the flow stress of the composite, for this mechanism, is given by:

$$Y_{\text{Uniaxial Compression}} = \frac{2G}{1-\upsilon}\sqrt{\frac{\theta b}{e\pi\lambda_m}} \qquad \text{(P6-2)}$$

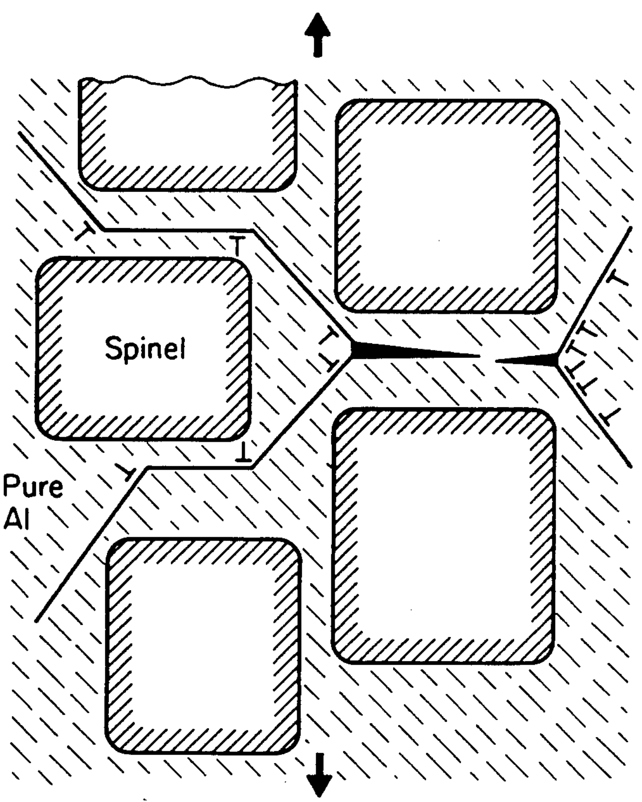
where Y is the flow stress in uniaxial compression, G is the shear modulus, υ Poisson Ratio, θ is the angle of the low angle boundary

Figure 1(a) and 1(b)



(a)
Uniaxial Compression
(Plastic Flow)

(b)
Uniaxial Tension
(fracture)



discussed above, b is the Burgers vector, and λ_m is the metal ligament size.

For the tension case, the pile up of dislocations leads to fracture, and the following equation obtains:

$$\sigma^{\text{fracture}}_{\text{uniaxial tension}} = \sqrt{\frac{4G\gamma_s}{\pi(1-\upsilon)} \cdot \frac{1}{\lambda_m}} \qquad \text{(P6-3)}$$

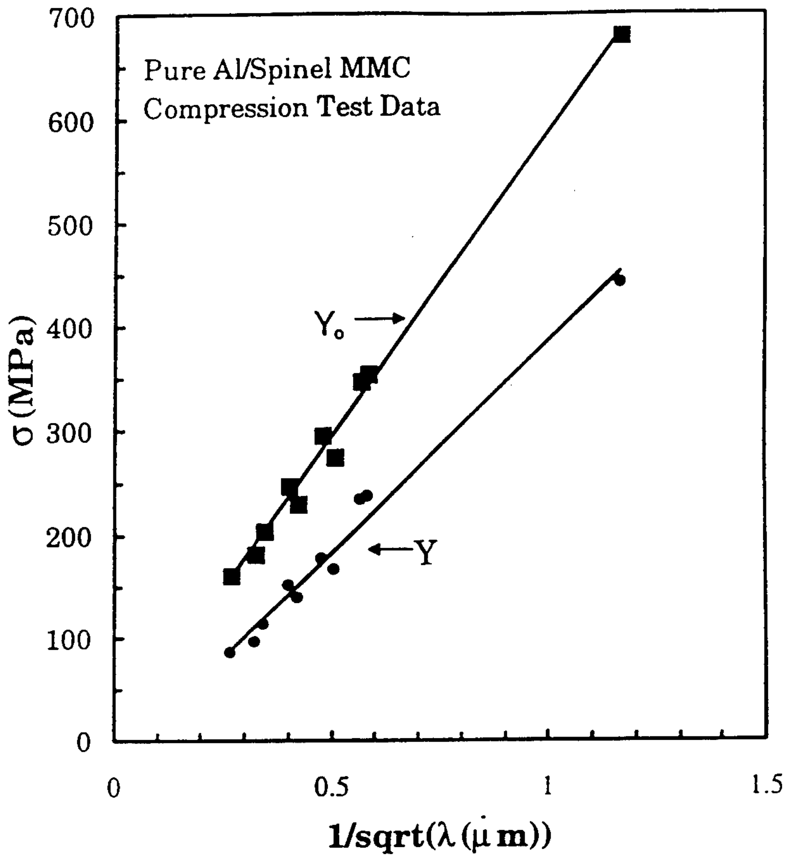
where γ_s is the surface energy.

Note that both equations predict an inverse square root dependence on λ_m, the metal ligament size, although the magnitude of the stress for compressive flow will be larger than for tensile fracture.

The above predictions were compared to data obtained from composites made with aluminum and spinel (magnesium aluminate) particles. Specimens were prepared in such a way that λ_m changed from less than 1μm to 15μm. The results are shown in Figs 2(a) and 2(b). The data show an excellent fit to the predicted inverse square root dependence on λ_m.

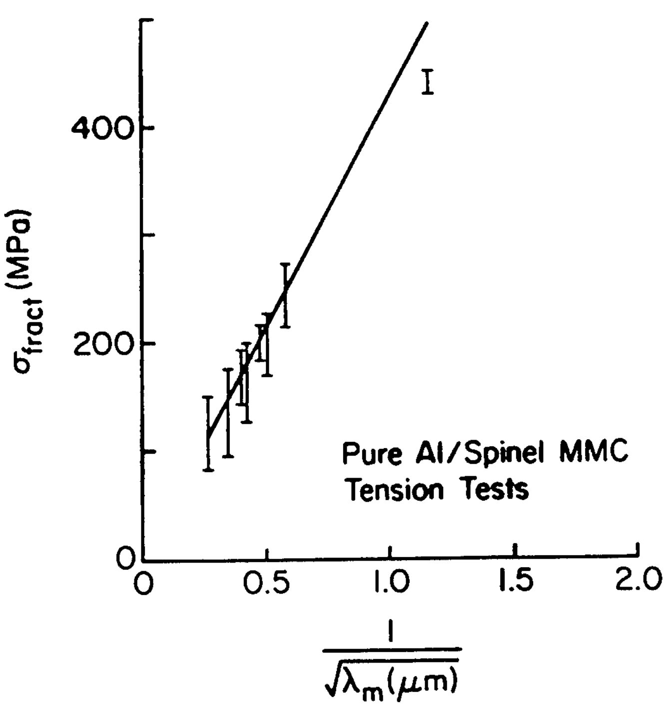
The equations given above also allow us to compare the magnitude of the measured flow stress, in compression and fracture stress, in tension, with data given in Figs 2(a) and 2(b). We have shown that the tensile fracture stress data has an excellent agreement with the values predicted by Eq. (P6-3). However, the magnitude of the flow stresses calculated from Eq. (P6-2) are about a factor of 6 smaller than the measured values. This

Figure 2(a) and 2(b)



(a)
Compression

(b)
Tension



difference is ascribed to the fact that the local von-Mises stress in the metal ligaments is lower than the applied stress due to the hydrostatic constraint imposed by the rigid ceramic particles.

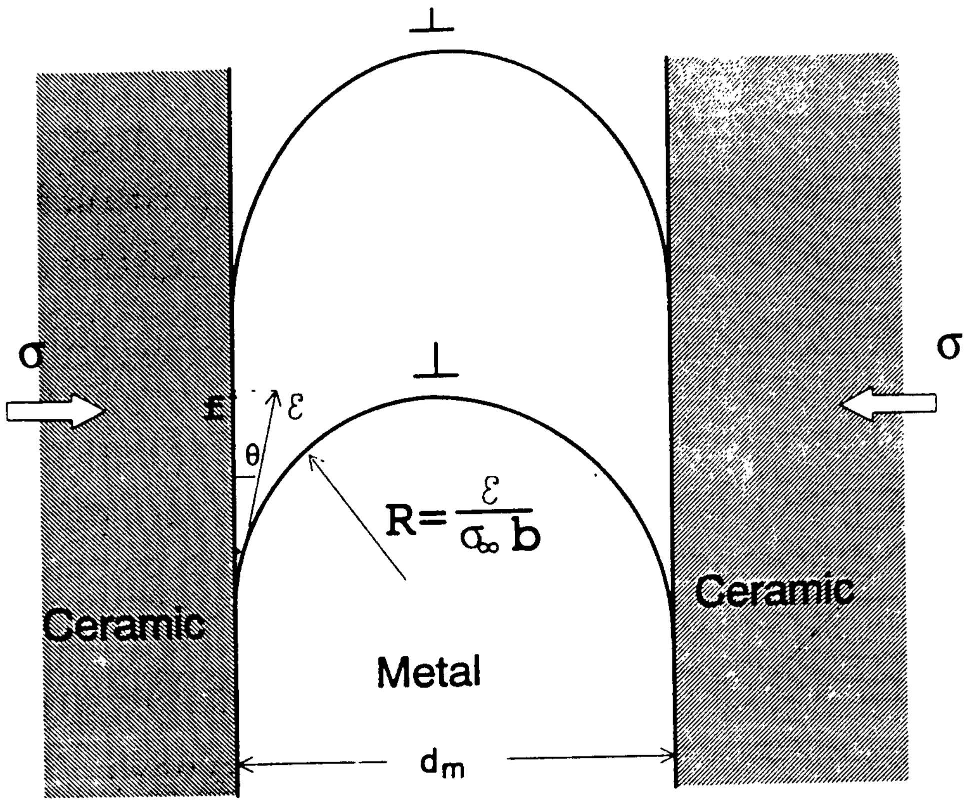
(a.2) CREEP RESISTANCE OF METAL-CERAMIC COMPOSITES

In high temperature creep the slip compatibility issue becomes superseded by recovery, that is the annihilation of dislocations promoted by dislocation climb. However, the dislocations can still become pinned at metal-ceramic interfaces. Our experimental work with composites made from niobium and alumina have led us to propose that the flow stress of the composite is controlled by the mechanism illustrated in Fig. 3. The flow stress depends on the rate of movement of dislocation which can be controlled either by its climb rate near the middle of the flow channel or by the drag of the dislocation along the metal-ceramic interface. This mechanism leads to the following equation for the flow stress:

$$\sigma = \frac{2\mathscr{E}}{d_m b} \cdot \cos\theta(\dot{\varepsilon}_r) \qquad \text{(P4-1)}$$

Here $\mathscr{E}$ is the line tension of the dislocation, $d_m=\lambda_m$, $\dot{\varepsilon}_r$ is the strain rate, and θ is the angle subtended by the dislocation at the boundary *while the dislocation is moving*. Note, that in this instance we find the flow stress is related inversely to the first power of the metal ligament size. This result highlights the importance of different mechanisms for deformation at ambient and high temperatures.

Figure 3



⊥
⊥
σ
σ
ε
θ
$R = \frac{\varepsilon}{\sigma_\infty b}$
Ceramic
Ceramic
Metal
d_m

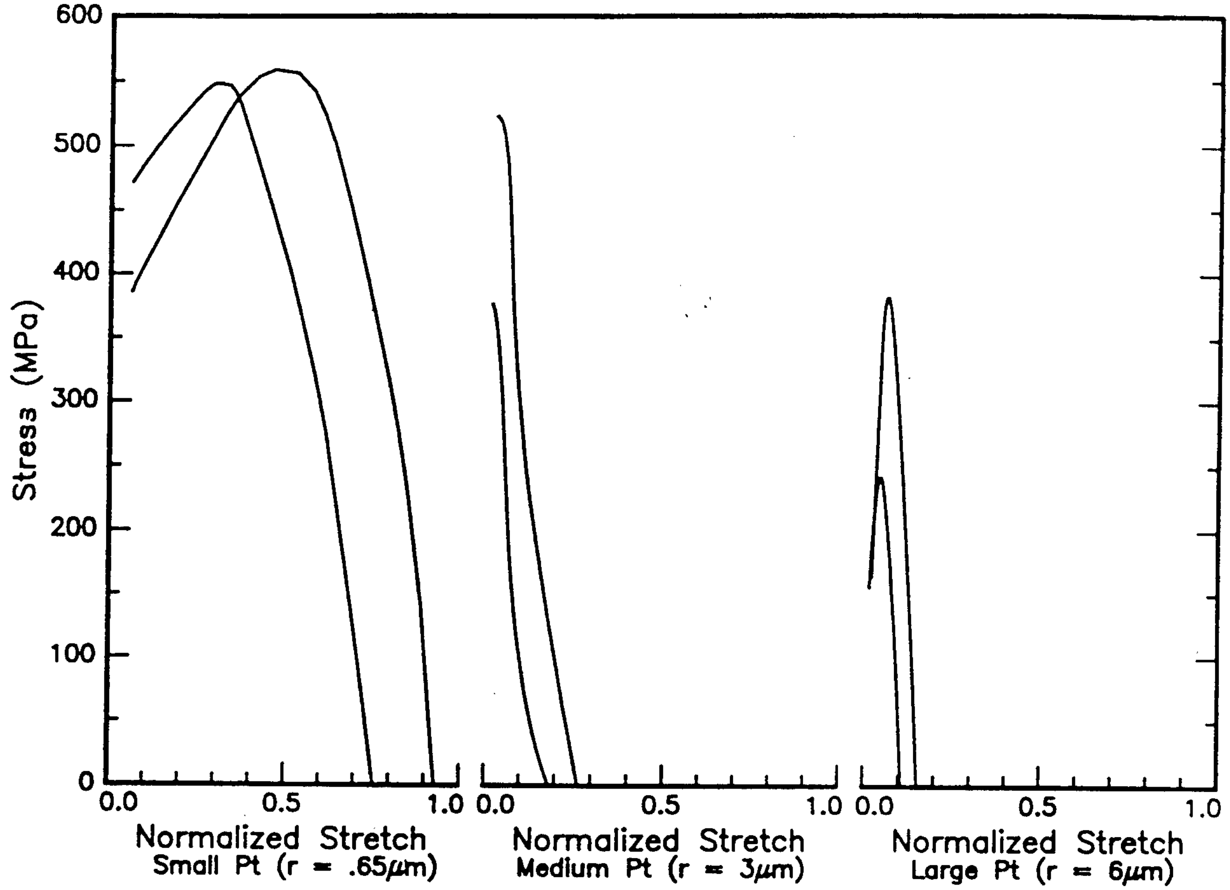
(a.3) DUCTILE TOUGHENING FROM THE METALLIC PHASE

The fracture toughness of metal matrix composites is largely determined by the work expended in the metallic phase during crack propagation. Thus it has long been recognized that the fracture toughness of the composite would increase with a higher volume fraction of the metal. Our work, however, has demonstrated that there is also a critical length scale of the metallic phase where there is a transition from a "brittle" to a ductile mode of fracture. The transition from ductile to brittle behavior occurs when the metal ligament size becomes greater than this critical value. Thus the contribution of the metallic phase to the fracture toughness (for a fixed volume fraction of the metal) is far greater when the metal ligament size is below this critical value.

The above concept has been demonstrated by innovative experiments where the *in-situ* stress strain curves of metal particles was measured in mode experiments. The normalized stress strain curves for metal particles having a radius of 0.65μm, 3.0μm and 6.0μm, obtained in this way are shown in Fig. 4. The area underneath the curves is the work done per unit volume of the metallic phase. Note the very large difference in the area for the small and the two large particle sizes (Ref. P2).

We have explained the above phenomenon by proposing a dislocation pile up mechanism for de-adhesion at metal ceramic interfaces. Plastic flow in the metal gives rise to dislocation pile-ups, and the stress concentration from these pile-ups induces

Figure 4



600
500
400
300
200
100
0
Stress (MPa)
0.0
0.5
1.0
Normalized Stretch
Small Pt (r = .65μm)
0.0
0.5
1.0
Normalized Stretch
Medium Pt (r = 3μm)
0.0
0.5
1.0
Normalized Stretch
Large Pt (r = 6μm)

fracture at the interfaces. In this way the criterion for brittle to ductile transition can be expressed in terms of the metal ligament size, the interfacial energy, and the applied stress. The ductile condition is obtained when:

$$\frac{\frac{\sigma_1}{3}\left(1+\frac{2\nu}{1-\nu}\right)\pi\left(1-\nu\right)\left[\frac{\sigma_1}{2}\left(1-\frac{\nu}{1-\nu}\right)-\tau_{Peierls}\right]d_m}{2G} \leq \Gamma_{M/C}$$

(P2-1)

where the pile-up length d_m is taken as the radius of the metal particle, G is the shear modulus of the inclusion, and σ_1 is the maximum in the tensile stress-strain curve for the average ductile particle. The interfacial energy term on the right is given by:

$$\Gamma_{M/C} = \left[\gamma_M + \gamma_C - \gamma_{M/C}\right] / 2 \qquad \text{(P2-2)}$$

Here γ_M is the surface energy of the metal, γ_C is the surface energy of the ceramic, and $\Gamma_{M/C}$ is the energy of the metal/ceramic interface.

The application of the above equation for different systems will lead to a critical value for d_m of about 1μm for strongly bonded metal-ceramic interfaces, to about 0.1μm for weakly bonded interfaces.

In reference P3 the above concepts were applied to the design of metal ceramic composites.

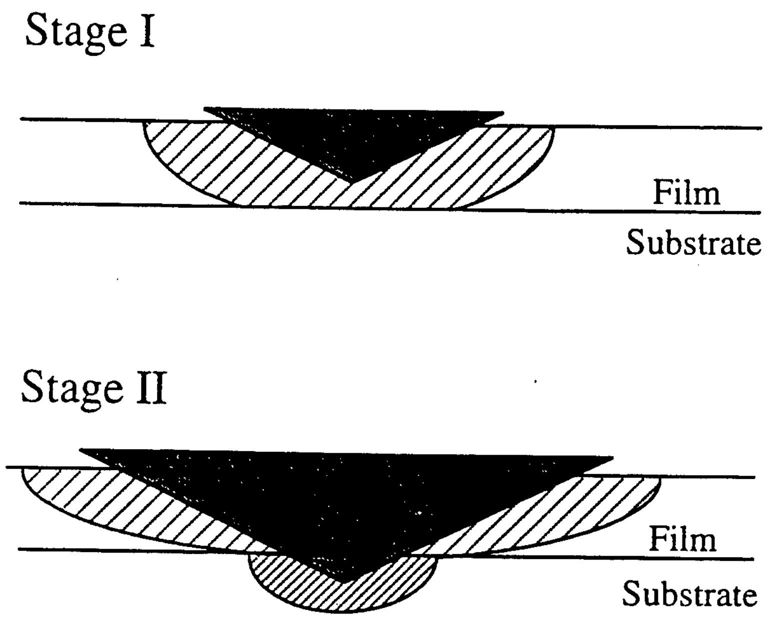
(b) Nanoengineering of Metal-Ceramic Interfaces:

The major accomplishment in this area is to show that insertion of an interlayer of titanium at copper/sapphire interface can significantly increases interfacial adhesion, and that increasing the thickness of the titanium layer from 0.7nm to 110nm does not result in a further benefit from the titanium layer.

The experimental approach in the study involved the development of two important new techniques. First, the copper and copper/titanium films were grown on sapphire in ultra-high-vacuum to ensure an atomically clean interface, by using a high temperature molecular beam epitaxial system. The metal overgrowths of the ceramic substrate were nearly singly crystal. The method and the results of this study are described in detail in Ref. P7.

The second important technique that we developed was the use of microindentation technique for estimating the interfacial strength. The procedure, the analysis for interpretation of data, as well as the detailed results are described in Ref. P6. Here, just the results of this study are summarized.

The microindentation experiments are divided into two Stages, as illustrated in Fig. 5. In Stage I, the Vickers indentor remains within the metallic overgrowth, and in Stage II the indentor pierces through the interface and penetrates into the ceramic substrate. For each stage the load vs. displacement curve is measured. Theoretical equations have been developed that interpret


Stage I
Film
Substrate
Stage II
Film
Substrate

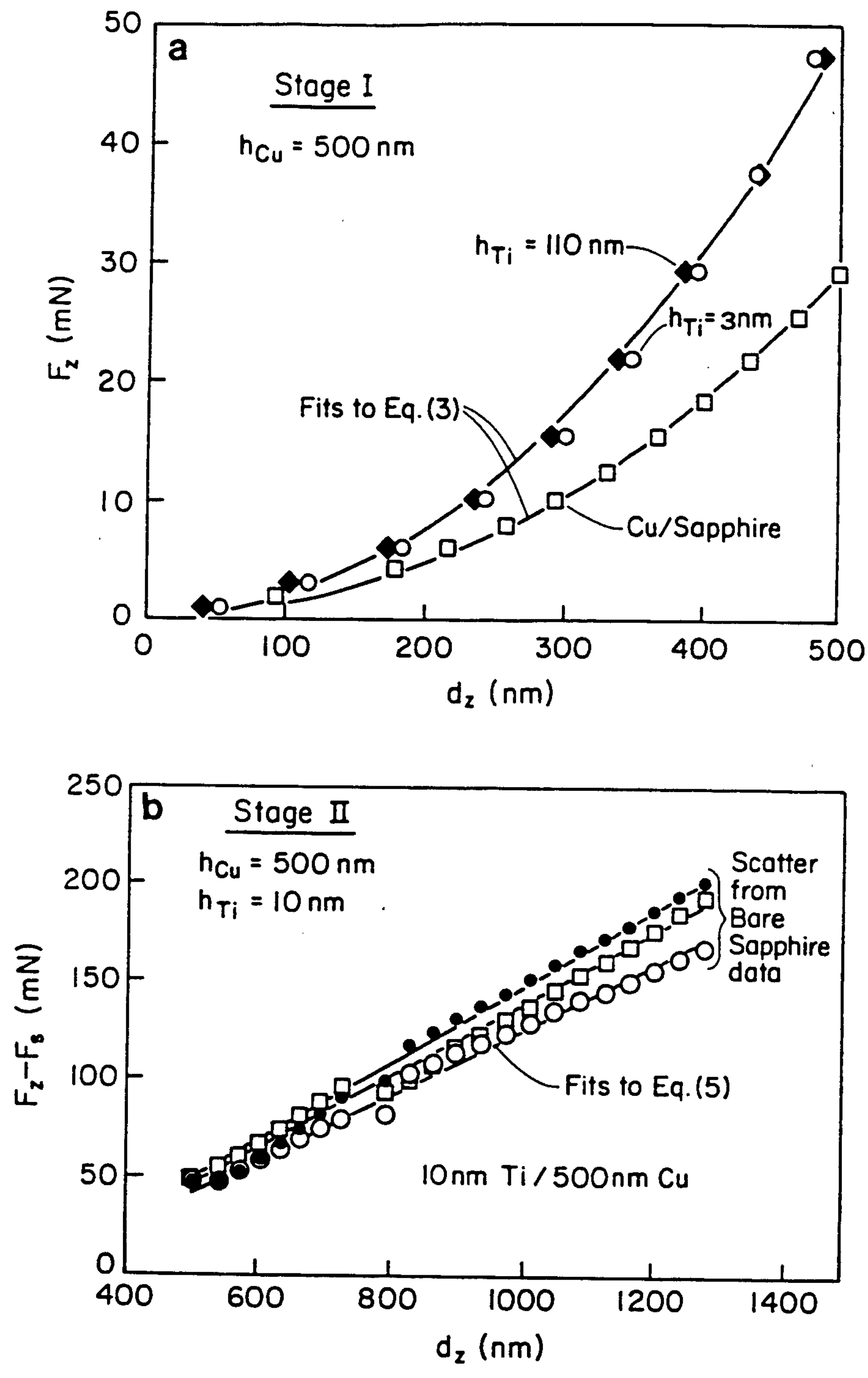
the data in terms of the interfacial shear strength. The experimental results and the fits to the data are shown in Fig. 6. The upper figure shows the Stage I data and the lower figure the Stage II data. The Stage I data is for three cases: copper on sapphire without Ti interlayer, and with titanium interlayers of thickness 3nm and 110nm. The Stage II data is for a 500 nm thick copper film and a 10nm thick Ti interlayer: the three sets of data for this case correspond to the scatter in the data obtained on bare sapphire. The solid lines are the predictions from the equations, which have the interfacial shear strength as the adjustable variable. The fit between theory and experiment then leads to an estimate of the interfacial strength.

In the Table below we give the estimates of the interfacial strength as a function of the thickness of the Ti interlayer. The thickness of the copper overgrowth was kept constant at 500nm.

Ti Interlayer	Stage I	Stage II
0 nm (Copper/Sapphire)	837 MPa	461 MPa
0.7 nm	1147	1236
3.0 nm	1410	1188
10.0 nm	1294	1319
110.0 nm	1391	1801

This piece of work shows that the strength of interfaces is dependent on the atomic level bonding across the interface.

Figure 6


a
Stage I
hCu = 500 nm
hTi = 110 nm
hTi = 3nm
Fits to Eq. (3)
Cu/Sapphire
Fz (mN)
dz (nm)
b
Stage II
hCu = 500 nm
hTi = 10 nm
Scatter from Bare Sapphire data
Fits to Eq. (5)
10nm Ti / 500nm Cu
Fz−Fs (mN)
dz (nm)

(d) Interfacial Thermal Boundary Resistance in Metal-Ceramic Composites.

In this paper (P8) we have developed a new way of understanding the resistance of interfaces to thermal conduction, and of applying these concepts to the design of metal-ceramic composites. Although the current work has been limited to particulate composites, the concepts can be easily applied to three dimensional interconnected microstructures as well as to multilayered laminates made from two or more constituents.

In Ref. P8 we have developed a parameter called the Kapitza radius, r_k, which is related to the thermal boundary resistance, R_{Bd}, as defined by Kapitza to describe the thermal resistance of interfaces between copper and liquid He, and the thermal conductivity of the matrix phase, K_m, by the equation:

$$r_k = R_{Bd}K_m \tag{P8-1}$$

From mechanistic point of view we have been able to relate r_k to the wavelength of the phonons at the Debye temperature, $\lambda_{ph}^{\theta_D}$, and η, which is the probability that phonons are transmitted from one lattice to the other lattice across the interface, as follows:

$$r_k = \frac{\lambda_{ph}^{\theta_D}}{\eta} \tag{P8-2}$$

The most interesting feature of the above expression is that r_k is likely to be similar for most interfaces. The reason is that the wavelength of the phonons at the Debye temperature is nearly the same for most materials (since conceptually the Debye

temperature is the defined by the condition that the phonons begin to be scattered by the lattice, that is, the phonon wavelength approaches the lattice parameter). The transmission efficiency of the phonons, η, will depend upon the "elastic stiffness" of the interfacial bonds: while this can differ from one bimaterial to another, we have little knowledge of this quantity. Nevertheless, if we assume that η does not change very significantly from one interface to another then, based upon the above discussion, we expect r_k to be a relatively constant quantity for different materials.

The Table, extracted from Ref. P8, gives measured values for r_k from six sets of experimental measurements. In this Table, K_m is the thermal conductivity of the matrix phase, K_d is the thermal conductivity of the dispersed phase, B is the ratio of these two quantities, v_f is the volume fraction of the dispersed phase, d is the particle size of the dispersed phase, α_0 is the thermal diffusivity of the composite in the asymptotic limit of very large particles (where the thermal conductivity becomes insensitive to the interfacial resistance), and K_0 is the thermal diffusivity of the composite in the large particle limit. The quantity in the last column is the Kapitza radius.

The striking result from this Table is that the values for the Kapitza radius do not differ from one bimaterial to another by more than a factor of 50%, which is in substantial agreement with Eq. (P8-2) above.

If the above result is indeed generally true, then it gives

an important guideline for the role of interfaces in the thermal conductivity of composite and laminated materials: *if the microstructural scale of the composite is less than about 1 μm then the thermal conductivity of the composite will be lower than the continuum limit, as a result of the contribution from the thermal boundary resistance of interfaces.*

System	K_m W/mK	K_d W/mK	B K_m/K_d	v_f (%)	d μm	α_0 mm^2/s	K_o W/mK	r_k μm
Al_2O_3(NiAl) This Study	27.2	78	0.35	33%	10.2-23.3	8.2		1.5±0.5
Al_2O_3(Ni) [9]	32	59.3	0.135	5%	0.10-2.20		34	1.0±0.5
Al(SiC) [11]	178	244	0.73	20%	0.70-28.0		215	≈ 1.0
Al(SiC) [12]	178	244	0.73	40%	0.70-28.0		225	1.5±0.5
ZnS(Diamond) [Every]	17.4	≈600	0.03	10%-30%	0.5-4.0	n/a	n/a	≈ 1.0

The references in the first column as given in P8.

The manner in which the thermal conductivity of the composite is influenced by interfaces is described in the following

equation:

$$\frac{K}{K_o} = \frac{\alpha}{\alpha_o} = 1 - 3v_f \frac{\frac{B-(1-2r_k/d)}{2B+(1+4r_k/d)} - \frac{B-1}{2B+1}}{1-3v_f\frac{B-1}{2B+1}} \qquad \text{(P8-3)}$$

The parameters in the above equation have been discussed in the context of the preceding table. Note that $K \rightarrow K_o$ as $d \rightarrow \infty$, that is the thermal conductivity approaches the continuum limit as the particle size becomes very large (where the thermal boundary resistance is assumed not to play any role). Plots of Eq. (P8-3) will show that the thermal conductivity drops significantly from the continuum limit when r_k/d becomes less than unity. Since r_k is apparently of order 1μm, the result suggests that the interfacial resistance will have a significant on the overall thermal conductivity when the microstructural scale of the structure becomes smaller than about 1μm.